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Factors Influencing the Use of Some Common Insecticide-Dispersing Agents

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INTRODUCTION

Incidental to a search for stickers for phenothiazine and nicotine peat, two solid insecticides notably poor in adhesive power, the occasion arose to investigate the efficiency of a number of common dispersing agents. Laboratory studies were therefore undertaken to determine the minimum quantities of various agents necessary for suspension stability, and from consideration of this together with other factors, such as versatility, cost, and stability against water hardness, heat, and bacteria, to arrive at a conclusion as to the best agent or group of agents to employ in later tests of stickers.

In this circular such terms as "dispersion," "suspension," and "emulsion," and their variations are frequently employed. "Dispersion" and "suspension" are equivalent and are used interchangeably; "emulsion," however, is applied only to suspensions consisting of oil, wax, or resin when dispersed in water. Dispersing agents include materials which, when added to a suspension, have the property of stabilizing the system; they become emulsifying agents only when they are used as one of the components in an emulsion.

DISPERSING AGENTS TESTED

Some well-known dispersing agents the properties of which are unaffected by water hardness were chosen for these tests. These materials are listed in table 1. The hydrogen-ion concentration in 1-percent solutions, as determined to a pH accuracy of ± 0.25 with

indicators, is given, because the effectiveness of many insecticides, such as rotenone¹ and phenothiazine, when held in suspension is influenced by the alkalinity or acidity of the aqueous medium. The stability of the agents against bacterial action, heating, and acids and the 1940 market prices are also included.

TABLE 1.—*Dispersing agents used, their hydrogen-ion concentration in 1-percent solutions, their stability against bacterial action, heat, and acids, and their price per pound*

Agent	Hydrogen-ion concentration	Stability	Price ¹
	<i>pH</i>		<i>Cents per pound</i>
Mineral:			
Bentonite, Wyoming.....	10.0	Very good.....	0.6
Kaolin.....	5.5	do.....	.4
Fuller's earth.....	9.0	do.....	.5
Vegetable:			
Whole-wheat flour.....	4.4	Poor.....	3
Karaya gum, No. 1.....	4.2	Fair.....	14
Arabic gum, amber.....	4.2	do.....	16
Saponin.....	9.0	do.....	145
Animal:			
Blood albumin, flake, dark.....	4.5	Very poor.....	13
Ammonium caseinate ²	8.0	do.....	14
Glue, bone, powdered.....	4.5	do.....	14
Whole-milk powder.....	6.5	do.....	16
Egg albumin, powdered.....	4.5	do.....	60
Synthetic:			
Alkyl diphenyl sodium sulfonate.....	8.5	Very good.....	43
Sodium lauryl sulfate.....	6.2	do.....	53

¹ Prices quoted are the minima for wholesale lots quoted in Oil, Paint, and Drug Reporter, Jan. 15, 1940.

² To prepare ammonium caseinate 1.3 cc. of 28-percent ammonium hydroxide per 10 gm. of casein was added to bring the pH to 8.0.

The stability of the dispersing agents was found to be important. Repeated experience showed that the mineral and synthetic agents needed no preservatives, but agents of the vegetable and, especially, the animal groups were susceptible to putrefaction. It was found feasible to preserve the vegetable agents by adding up to 70 mg. of formaldehyde per gram of agent. Gum arabic, gum karaya, and saponin gave no indication of decomposition or loss of dispersing property when formaldehyde was added, but whole-wheat flour did show loss of dispersing power in time. It was concluded, therefore, that a chemical change occurred even in the absence of bacterial action, and that it was not practical to keep the flour solutions from day to day. The agents of animal origin were attacked very quickly, usually within 24 to 48 hours, with development of a foul odor and changes in appearance. No satisfactory preservative was found for this class of agents; copper sulfate and formaldehyde, which had the best preservative properties of those tried, were either ineffective or reacted with the agents, lessening their dispersing efficiency. Therefore, the animal agents were made into solution as needed.

OTHER MATERIALS USED

The foregoing dispersing agents were tested in suspension with five materials commonly used in insecticides—petroleum oil, menhaden fish oil, rosin residue, paraffin, and phenothiazine.

¹ TURNER, NEELY. NOTES ON ROTENONE AS AN INSECTICIDE Jour. Econ. Ent. 25:1228-1237, illus. 1932.

PETROLEUM OIL

The petroleum oil was a highly refined, water-white oil with the following specifications: Unsulfonatable residue 94 percent, viscosity (Saybolt at 100° F.) 105 seconds, refractive index at 20° C. 1.4669, specific gravity (20°/15° C.) 0.8507, solidification point -2° C., volatility (24 hours' heating at 100° F.) 1.5 percent, acid value 0, saponification number 0, iodine value (Hanus) 2, price 20 to 30 cents per gallon. This type of oil is used extensively against scale insects.

MENHADEN FISH OIL

The menhaden fish oil was a commercial-grade, dark-brown drying oil having the following properties: Viscosity (Saybolt at 100° F.) 182 seconds, specific gravity (20°/15° C.) 0.9276, solidification point -4° C., volatility (24 hours' heating at 100° F.) 0.2 percent, acid value 34.7, saponification number 190, iodine value (Hanus) 154. The price was 35 to 40 cents per gallon. This oil has been used principally as a sticker, but it possesses a limited scale-killing property.

ROSIN RESIDUE

Rosin residue, type 1, was a material of very high viscosity, dark-brown color, and rosin odor, which on spreading out in thin films hardened sufficiently in 48 hours to lose its tackiness. Goodhue and Fleming² have given the physical and chemical constants for this material, which is a byproduct in the manufacture of rosin. It is priced at 3 to 4 cents per pound in drum lots, and has had limited use as a sticker for certain insecticides.

PARAFFIN

To 3 parts by weight of melted paraffin 2 parts of benzene was added to give a product of wider solidification range than that of paraffin alone. This lowered the melting point of the paraffin from 52° C. to approximately 35°. The price of the refined paraffin was 4.5 cents per pound, that of the benzene 16 cents per gallon. Paraffin is the cheapest representative of the solid hydrocarbons. It has not yet gained much usage, but it holds promise as a protectant for organic insecticides that are subject to oxidation and photochemical decomposition under outside conditions.

PHENOTHIAZINE

The sample of phenothiazine used was a dark-green powder melting at 178.5° C. (melting point of pure compound 180°). A high degree of purity was thus indicated for the commercial sample, which was selling for 50 to 60 cents per pound. Phenothiazine has achieved limited importance as a stomach insecticide.

² GOODHUE, LYLE D., and FLEMING, WALTER E. STICKERS FOR DERRIS AS AN INSECTICIDAL SPRAY. Jour. Econ. Ent. 29: 580-583. 1936.

DILUTION WATER

All suspensions were prepared with water made hard in the laboratory³ by dissolving 1.75 gm. of calcium chloride (CaCl_2), 1.43 gm. of magnesium sulfate ($\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$), and 0.87 gm. of sodium bicarbonate in 10 liters of distilled water. The total hardness of this water toward the soap was 240 parts per million, calculated as CaCl_2 , as compared with about 200 parts per million for water of average hardness. The pH value as determined with cresol red and phenolphthalein indicators was between 8.2 and 8.8.

DISPERSION METHODS

The methods of dispersion depended on the properties of the material to be dispersed. For the two oils 5 cc. was placed in a 1-liter glass-stoppered cylinder, the aqueous phase containing the dispersing agent added in amount to give a total volume of 1 liter, and the mixture stirred for 2 minutes with a high-speed drink mixer. The concentration of the oil in the emulsion was thus 0.5 percent, and emulsification was carried out at the ultimate spray dilution under conditions similar to those obtained under agitation in the tank of a spray machine. The stability or break-down of the emulsion was apparent immediately on mixing, whereas when oil is dispersed in the form of a concentrated emulsion followed by dilution more stirring is required before equilibrium is reached. It was also found that the minimum concentration of emulsifying agent to give a stable emulsion at a high oil-water ratio was often quite distinct from that required at a low oil-water ratio.

Rosin residue and paraffin were emulsified in the molten condition, and the emulsion so formed was cooled to room temperature, with stirring, according to well-known methods.⁴ The apparatus consisted of a high-speed drink mixer and a 15- by 3.5-cm. Pyrex test tube. The two-button shaft of the mixer extended nearly to the bottom of the tube, and both were held tightly in place by clamps. By means of a heated pipette 5 cc. of the molten material was transferred to the Pyrex tube containing 50 cc. of an aqueous solution of the emulsifying agent. The contents were heated nearly to the boiling point of the water and then stirred for 2 minutes. The mixing tube was then cooled with water, with continued agitation, until the contents reached room temperature. Dilution was made at this point to give a total volume of 1 liter, the rosin or paraffin content being 0.5 percent. About 6 minutes was required for emulsification and cooling. If foaming was excessive, the concentration of the emulsifying agent was reduced to such a point as would produce a minimum of foam and yet bring about emulsification, and the remainder added before dilution.

Suspensions of phenothiazine were prepared by wetting 5 gm. of the powder with 5 cc. of 45-percent ethyl alcohol, then adding the dispersing agent, usually as a 1-percent solution, and finally diluting to 1 liter. This mixture was stirred with the drink mixer for 1

³ GINSBURG, JOSEPH M. EFFECT OF DIFFERENT SOAPS ON FORMATION OF SOLUBLE ARSENIC FROM LEAD ARSENATE IN SOFT AND HARD WATERS. *Jour. Econ. Ent.* 30: 583-590. 1937.

⁴ BENNETT, H., editor-in-chief. *THE CHEMICAL FORMULARY* . . . v. 1, pp. 151-164. Brooklyn, N. Y. 1934.

minute and then allowed to stand for observation. A concentration of 5 gm. per liter is equivalent to slightly more than 4 pounds per 100 gallons, which is frequently employed in experimental orchard spraying with phenothiazine.

TESTS OF DISPERSION EFFICIENCY

The relative efficiency of the various agents in dispersing each material was tested by determining the minimum quantity necessary to produce a stable suspension. For each material several concentrations of the dispersing agent were tried until a range was found below the lower limit of which it was definitely unstable and above which it was definitely stable. It was not feasible to locate the exact concentration producing critical stability, because suspension breakdown was not always sharply defined.

The oil emulsions and those of rosin residue and paraffin were considered stable if no coagulation or fusion of droplets was evident after standing for 30 minutes. They were classed as unstable if, after settling, the accumulated mass of particles was not redispersible on shaking, or if the mass adhered to the sides of the container. Phenothiazine suspensions were assumed to be stable when the air occluded during dispersion was not sufficient to cause the particles to conglomerate at the surface of the aqueous medium.

The results of these tests are summarized in table 2, together with the unit cost of the dispersing agent when in suspension in the upper limit of concentration given.

TABLE 2.—*Efficiency of dispersing agents in holding various materials in suspension, together with their cost*^{1 2}

Agent	Petroleum oil		Menhaden fish oil		Rosin residue		Paraffin		Phenothiazine	
	Quantity of agent for stable suspension	Cost per 100 gallons	Quantity of agent for stable suspension	Cost per 100 gallons	Quantity of agent for stable suspension	Cost per 100 gallons	Quantity of agent for stable suspension	Cost per 100 gallons	Quantity of agent for stable suspension	Cost per 100 gallons
	<i>Mg. per liter</i>	<i>Cts.</i>	<i>Mg. per liter</i>	<i>Cts.</i>	<i>Mg. per liter</i>	<i>Cts.</i>	<i>Mg. per liter</i>	<i>Cts.</i>	<i>Mg. per liter</i>	<i>Cts.</i>
Blood albumin.....	0.4-0.8	0.01	10-20	0.22					10-20	0.22
Egg albumin.....	.8-1.6	.08	10-20	1.0					6-12	.60
Ammonium caseinate.	2.5-5.0	.05	20-40	.46	15-30	0.36	20-80	0.94	4-8	.10
Glue.....	2.0-4.0	.04	160-320	3.7	200-400	4.7	400-800	9.4	7-14	.17
Saponin.....	15-30	3.6	100-200	24			150-200	24	30-40	4.8
Arabic gum.....	20-40	.54	700-900	12	500-1,000	13	120-250	3.3	10-20	.26
Whole-milk powder.	5-10	.14	100-200	2.7	750-1,250	17	1,000-2,000	27	30-60	.80
Whole-wheat flour.	20-40	.10	500-800	2.0			1,000-2,500	6.3	8-16	.04
Karaya gum.....	50-100	1.2	800-1,000	12					30-50	.58
Bentonite.....	1,500-3,000	1.5	20-40	.02					70-90	.05
Fuller's earth.....	1,500-3,000	1.3	400-600	.25						
Sodium lauryl sulfate	2,000-4,000	177	1,000-1,300	58			1,000-1,500	66	40-80	3.5
Alkyl diphenyl sodium sulfonate.	2,000-4,000	147	1,100-1,300	47			500-1,000	36	160-320	12
Kaolin.....	800-1,600	.53	3,000-5,000	1.7						

¹ The concentration of all the materials held in suspension is 0.5 percent, by weight in the case of phenothiazine and by volume in all other cases.

² Costs are given for maximum quantity of dispersing agent in the range given.

Foaming during the process of dispersion is objectionable, because the foam tends to deprive the particles of sufficient agent for colloidal protection, especially where there are limited quantities of the dispersing agent to begin with. It also sometimes causes the liquid to run over the top of the mixing tube. Tests to determine whether the foaming tendency of solutions of the different dispersing agents could be correlated with their dispersing power gave negative results. No practical connection was found between the two properties, further than the observation that the best dispersing agents were also good foaming agents. There was no correlation between the dispersing efficiency and either the volume of foam produced or its persistence.

RELATIVE EFFICIENCY OF DISPERSING AGENTS WITH SPECIFIC MATERIALS

When the results of these suspension-stability tests (table 2) were considered together with the factors of cost and the stability of the dispersing agent against water hardness, heat, and bacteria (table 1), the following conclusions were drawn as to their utility with specific materials, under the conditions of the experiments.

For petroleum emulsions blood albumin, glue, ammonium caseinate, egg albumin, whole-wheat flour, and whole-milk powder are economical in the order named, provided the mixtures are to be used before putrefaction sets in. If the emulsions are to be kept several days before use, arabic gum, karaya gum, kaolin, and bentonite are applicable and economical in the order named, either because they are not susceptible to bacterial decomposition or because the use of preservatives is feasible. With the emulsifying efficiency shown, saponin is relatively too expensive to be of practical value. The synthetic materials alkyl diphenyl sodium sulfonate and sodium lauryl sulfate not only showed the poorest efficiency of all the agents tested, but they are also very high priced.

With menhaden fish oil the clays, which include bentonite, fuller's earth, and kaolin, appear to be the most promising group of emulsifiers, because they possess efficiency, resistance to putrefaction, ease in handling, and low cost. Bentonite is the most economical of all the agents tested. Saponin, sodium lauryl sulfate, and alkyl diphenyl sodium sulfonate tended to produce reversed emulsions on continued agitation and are therefore not considered reliable for use with menhaden oil. They are also impractical on the ground of high price or poor efficiency, or both. Since this oil contained large amounts of free fatty acid and the dilution water was appreciably alkaline, soap formation may have influenced the stability of some of the emulsions, though in which direction it is hard to say. The pH value of the emulsions ranged from 6.5 to 8.0.

Rosin residue was, in general, difficult to emulsify, only 4 agents out of the 14 tested being capable of emulsifying it—ammonium caseinate, glue, arabic gum, and whole-milk powder. The pH value of these emulsions was about 7.5. They were especially sticky, owing to the high viscosity of the rosin residue, and it was impossible to reemulsify coagulated masses simply by shaking the container. Stability of particles was therefore especially important. In this respect whole-milk powder outranks the other agents, and under the experi-

mental conditions is rated as the best temporary emulsifier for rosin residue. All the agents are susceptible to putrefaction, but since arabic gum is preservable with formaldehyde, it appears to have the greatest utility.

With paraffin only eight of the agents were able to bring about complete dispersion and stability in the suspensions. Of the agents susceptible to decomposition, the order of utility was ammonium caseinate, whole-wheat flour, glue, and whole-milk powder. Of the agents that were either stable or preservable from putrefaction, arabic gum, saponin, alkyl diphenyl sodium sulfonate, and sodium lauryl sulfate were economical in the order named. Arabic gum was the most practical dispersing agent in either class because of its emulsifying efficiency, resistance to putrefaction, cleanliness, and ease of handling. The paraffin was neutral and could not have affected the pH value of the emulsion.

In the tests with phenothiazine all the dispersing agents but kaolin and fuller's earth gave stable suspensions. The pH value of the karaya-gum suspension was 7.5, and with all the other agents it was between 8.0 and 8.5. The remaining clay, bentonite, showed fairly good dispersive power, but caused decomposition of the phenothiazine, as noted by formation of a brick-red color, which was accelerated by acidification of the suspension. The component of bentonite responsible for the change was not discovered. The clays were therefore ruled out for use with phenothiazine. Of the dispersing agents susceptible to bacterial decomposition, whole-wheat flour, ammonium caseinate, glue, blood albumin, egg albumin, and whole-milk powder were economical in the order named. Of the stable or preservable agents, arabic gum, karaya gum, sodium lauryl sulfate, saponin, and alkyl diphenyl sodium sulfonate were economical in the order named, the two gums having a decided advantage. Arabic gum is considered to be the most useful agent for dispersion of phenothiazine, because it is efficient, preservable, clean, easy to handle, and cheap.

GENERAL DISCUSSION OF RELATIVE EFFICIENCY OF AGENTS

When broadly considered in relation to the ease with which they were dispersed, refined petroleum oil, phenothiazine, menhaden fish oil, rosin residue, and paraffin rank in the order named (table 2). Approximately the same dispersing-agent order was maintained for all these materials, where dispersion was carried out with approximately equal quantities.

The albumins were generally most efficient, but they were limited to use with materials that might be dispersed in the cold. Heating the albumins in attempts to emulsify rosin residue and paraffin produced coagulation as in the poaching of an egg, and even in the case of oils the utility of the albumins was generally limited because of susceptibility to bacterial decomposition. Blood albumin is the more practical of the two because of its lower price.

Of the 14 agents tested only 4 were capable of dispersing all the materials—ammonium caseinate, glue, arabic gum, and whole-milk powder—and they were efficient in the order named. The utility of ammonium caseinate was limited, however, because of its chemical

instability toward acids, acid salts, and bacteria. Glue and whole-milk powder were also affected by heat or bacteria, or both. On the contrary, arabic gum was relatively stable. Versatility, together with moderate price and efficiency, places arabic gum as the best all-around dispersing agent for general use.

As a group the animal products have the highest dispersing efficiency, but their utility is limited because they lack stability. This group is generally most economical when the sprays are to be used immediately after mixing. The products of vegetable origin are not so efficient as the animal products, but are stable against heat, preservable against bacteria, and possess little objectionable odor. With the exception of saponin, they appear to be economical. The mineral agents are extremely cheap and stable but relatively inefficient as dispersing agents. Bentonite in emulsification of fish oil, however, showed high dispersion efficiency and proved to be the most promising agent to use with this material. The clays were limited in their applicability, being practical in dispersing only the petroleum and fish oils. Alkyl diphenyl sodium sulfonate and sodium lauryl sulfate, composing the synthetic group, were capable of dispersing four out of the five materials tested, and they possessed stability, but their low general efficiency and relatively high prices make them undesirable as dispersing agents.

SUMMARY

Certain factors considered as affecting the efficiency of insecticidal dispersing agents—namely, dispersing efficiency, cost, stability against water hardness, heat, and bacteria, and versatility in dispersing different kinds of material—were investigated in the laboratory for 14 commonly used agents. Dispersing efficiency was gaged by the minimum quantity of dispersing agent necessary to produce a stable suspension under a selected set of conditions. An agent was classified as stable if it was unaffected by heat, acids, and bacteria, or if it was preservable by artificial means.

These dispersing agents were tested with five materials commonly used in insecticides—refined petroleum oil, phenothiazine, menhaden fish oil, rosin residue, and paraffin.

The best all-around agent was arabic gum, because it possessed efficiency, low cost, resistance to decomposition, versatility, cleanliness, and ease in handling.

In the unstable class, which was composed largely of agents of animal origin, blood albumin, egg albumin, ammonium caseinate, glue, and whole-milk powder were generally more efficient than members of other groups. Their chief usefulness appeared to lie in the preparation of insecticidal sprays which are to be used within 24 hours after mixing.

Only 4 out of the 14 agents studied were capable of dispersing all the materials. These 4 versatile agents were ammonium caseinate, glue, arabic gum, and whole-milk powder, efficient in the order named.

In the emulsification of refined petroleum oil, blood albumin was the best agent in the unstable class and arabic gum was the best agent in the stable class. For menhaden fish oil, bentonite clay was the best agent regardless of class. For rosin residue, whole-milk

powder was the best agent in the unstable class, arabic gum the best agent in the stable class. In the dispersion of paraffin, ammonium caseinate was the best agent in the unstable class, arabic gum the best in the stable class. For phenothiazine, whole-wheat flour was the best agent in the unstable class, arabic gum the best in the stable class.

No correlation of practical value was found between the general dispersing efficiency of the agents and their tendency to foam.

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